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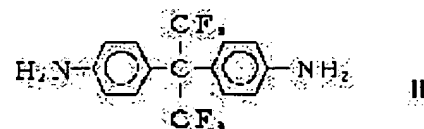
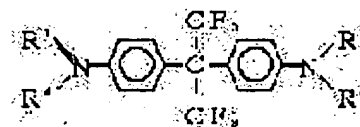
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(54) TERTIARY AMINE COMPOUND HAVING FLUOROCARBON GROUP

(57)Abstract:

PURPOSE: To obtain a new tertiary amine compound, having a fluorocarbon group, capable of satisfying all the fundamental electrophographic characteristics and useful as a photoconductive material.

CONSTITUTION: A compound expressed by formula I (R1 and R2 are H, substitutable alkyl or substitutable aryl, except that both are simultaneously H), e.g. 2,2-bis(4-N,N-dimethylaminophenyl)hexafluoropropane. The compound expressed by formula I in which R1 and R2 are (substituted)alkyl is obtained by reacting 2,2-bis(4-aminophenyl)hexafluoropropane expressed by formula II with an alkyl halide, a dialkyl sulfate, etc.



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* NOTICES *

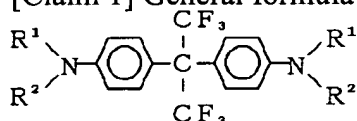
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CLAIMS

[Claim(s)]

[Claim 1] General formula [** 1]



(The inside R1 and R2 of a formula expresses the aryl group which is not permuted [the alkyl group which is not permuted / a hydrogen atom, a permutation, or /, a permutation, or], and even if respectively the same, it may differ.) However, R1 and R2 remove to coincidence the case where it is hydrogen. Tertiary-amine compound which has the fluorocarbon radical expressed.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the tertiary-amine compound which has a new fluorocarbon radical useful as an organic photoconductivity ingredient for electrophotography.

[0002]

[Description of the Prior Art] Conventionally, as an organic photoconductivity material of the photoconductor used in an electrophotography method, much proposals of Poly N-vinylcarbazole, a triphenylamine compound (U.S. Pat. No. 3,180,730), a benzidine compound (the 3,265,496th JP, 39-11546,B of an United States patent, JP, 53-27033,A), etc. are made, for example.

[0003] The "electrophotography method" said here is one of the image formation methods which acquire an electrostatic latent image, visualize this latent-image section with the development means using a toner etc. further, and formed the image by making it the photoconductor of a photoconductivity first charged by corona discharge etc. in a dark place, performing image-like exposure subsequently generally, and making the charge of the exposure section discharge alternatively. Being charged in suitable potential in one dark place as a fundamental property required of the photoconductor in such an electrophotography method, that there is little discharge of the charge in two dark places, discharging a charge promptly by 3 light exposure, etc. are mentioned.

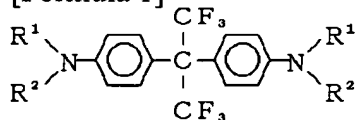
[0004]

[Problem(s) to be Solved by the Invention] However, the actual condition is that the conventional photoconductivity organic material has not necessarily satisfied these demands. Therefore, the purpose of this invention satisfies all fundamental electrophotographic properties, and is to offer the tertiary-amine compound which has a new fluorocarbon radical useful as a photoconductivity ingredient.

[0005]

[Means for Solving the Problem] according to this invention -- the following general formula (** 1) --

[Formula 1]



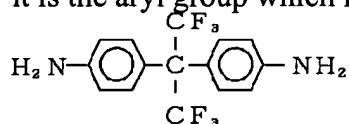
(The inside R1 and R2 of a formula expresses the aryl group which is not permuted [the alkyl group which is not permuted / a hydrogen atom, a permutation, or /, a permutation, or], and even if respectively the same, it may differ.) However, R1 and R2 remove to coincidence the case where it is hydrogen. The tertiary-amine compound which has the fluorocarbon radical expressed is offered.

[0006] In said general formula (** 1), a phenyl group, a halogen atom, an alkoxy group, a FENIRU oxy-radical, etc. are mentioned as an alkyl group as a substituent in low-grade alkyl groups, such as a methyl group, an ethyl group, a propyl group, and butyl, and a permutation alkyl group. On the other hand, as an aryl group, a phenyl group, a naphthyl group, an anthryl radical, a fluorenyl group, an ASENAFU thenyl radical, a pyrenyl radical, a phenan tolyl group, a chestnut SENIRU radical, a fluoran thenyl

radical, etc. are mentioned, and, as for these aryl groups, you may have a low-grade alkyl group, a lower alkoxy group, an alkylene dioxy radical, a permutation amino group, and a halogen atom as a substituent.

[0007] The tertiary-amine compound which has the fluorocarbon radical shown by said general formula 1 concerning this invention is the new matter, and when R1 and R2 are the alkyl groups which are not permuted [a permutation or], it can be easily manufactured using the alkyl halide which is the alkylating agent of nitrogen, the permuted alkyl halide, a dialkyl sulfuric acid, a sulfonate, etc.

[0008] They are the primary amine compound shown by the following-ization 2 on the other hand when it is the aryl group which is not permuted [a permutation or] in both R1=R2, and [Formula 2].



It can manufacture by making aryl halide react in the temperature of about 150-250 degrees C under a nitrogen air current in a non-solvent or a solvent with copper powder, copper oxide or halogenation copper, and alkali salt.

[0009] In this case, as alkali salt, a sodium hydroxide, a potassium hydroxide, a sodium carbonate, potassium carbonate, etc. can be mentioned. Moreover, as a reaction solvent, a nitrobenzene, dichlorobenzene, a quinoline, N.N-dimethylformamide, dimethyl sulfoxide, N-methyl pyrrolidone, 1,3-dimethyl-2-imidazolidinone, etc. can be mentioned.

[0010] Moreover, when it is the aryl group which is not permuted [a permutation or] in both R1 !=R2, after protecting the primary amine compound of a general formula (** 2) by an acyl group etc., it is made to react with aryl halide, and can manufacture by hydrolyzing subsequently and performing a reaction with aryl halide again. The conditions at the time of reacting with aryl halide are the same as that of what was shown previously.

[0011] It is obtained by making the alkylating agent of hydrolyzing [1 / R1 performed the reaction with aryl halide by having used the above-mentioned N-acyl compound as the start ingredient, when R2 was the aryl group which is not permuted / a permutation or /, and]-by alkyl group which is not permuted [a permutation or], nitrogen react.

[0012] The tertiary-amine compound which has the new fluorocarbon radical obtained by this invention is very useful as a photoconductivity material in the photo conductor for electrophotography, and sensitization is carried out optically or chemically by sensitizers, such as a color and Lewis acid. Furthermore, this thing divides as charge transportation matter in the so-called functional discrete-type photo conductor which uses an organic pigment or an inorganic pigment as the charge generating matter and is useful.

[0013] as the above-mentioned sensitizer -- thiazine dye [, such as xanthin colors, such as thoria reel methane colors, such as Methyl Violet and a crystal violet, a rose bengal, erythrosine, and a rhodamine and a methylene blue,], 2 and 4, and 7-trinitro-9-full -- me -- non, 2, and 4-dinitro-9-full -- me -- non etc. is mentioned.

[0014] As an organic pigment, moreover, C.I. pigment blue 25 (CI No.21180), Azo pigment, such as the C eye pigment red 41 (CI No.21200) and the C eye pigment red 3 (CI No.45210), Phthalocyanine pigment, such as C.I. pigment blue 16 (CI No.74100), indigo system pigments, such as C eye BATTOBURAUN 5 (CI No.73410) and C.I. vat dye (CINO.73030), and Argo -- a scaw -- perylene system pigments, such as Let B and indanthrene Scarlett R, are mentioned. Moreover, inorganic pigments, such as a selenium and selenium-tellurium, a cadmium sulfide, and alpha-silicon, can also be used.

[0015]

[Effect of the Invention] The tertiary-amine compound which has the new fluorocarbon radical shown by said general formula (** 1) concerning this invention From functioning as having described above effectively as a photoconductivity material, and sensitization being carried out optically or chemically

by sensitizers, such as a color and Lewis acid It is useful as charge transportation matter in the so-called functional discrete-type sensitization layer which was suitably used as charge transportation matter of the sensitization layer of the photo conductor for electrophotography etc., and classified the charge generating layer and the charge transportation layer into the bilayer especially.

[0016]

[Example] Hereafter, an example explains this invention to a detail.

Example 1 (general formula (** 1); R1=R2= composition of CH3)

2 and 2-bis(4-aminophenyl) hexafluoropropane 6.69g was dissolved in tetrahydro furan 65ml, to this, subsequently, 4.80g of 60% sodium hydride was added, and 17.0g of methyl iodides was agitated for it at 45-50 degrees C for 5 hours. After cooling radiationally to a room temperature, 500ml of saturation brine was filled with contents, and it extracted with toluene. After drying a toluene layer with the magnesium sulfate after rinsing, the solvent was distilled off under reduced pressure, and 5.86g of light yellow crystals was obtained. With activated carbon, it recrystallized [methanol] and the pure article of 2 of a non-color plate-like crystal and 2-bis(4-N and N-dimethylamino phenyl) hexafluoropropane was obtained.

Melting point 114.5-115.5 degrees C Elemental-analysis value (%)

C H N Fruit ** Value 58.55 5.11 7.16 C19H20F6N2 ***** -- calculated value 58.45 5.17 7.18 -- the infrared-absorption-spectrum Fig. (KBr briquette method) of this compound is shown in drawing 1 .

[0017] Example 2 (general formula (** 1); R composition of 1=R2=C6H5CH2-)

The benzyl bromide was used instead of the methyl iodide used in the example 1, and also it reacted according to the example 1, and the pure article of 2 and 2-bis(4-N and N-dibenzyl aminophenyl) hexafluoropropane was obtained as colorless prism-like **.

Melting point 168.5-169.5 degrees C Elemental-analysis value (%)

C H N Fruit ** Value 74.31 5.03 4.94 C43H36F6N2 ***** -- calculated value 74.33 5.23 4.03 -- the infrared-absorption-spectrum Fig. (KBr briquette method) of this compound is shown in drawing 2 .

[0018] Example 3 [general formula (** 1); synthetic] of R1=-H R2=-C6H4CH3 (p)

The heating reflux of 2 and 2-bis(4-acetamide phenyl) hexafluoropropane 8.36g and the p-BUROMO toluene 34.18g was carried out for bottom 14 hours of a nitrogen air current with 8.29g of potassium carbonate, and 0.30g of copper powder. After cooling radiationally to the room temperature and carrying out filtration removal of the insoluble section, the filtrate was dissolved in toluene, the solvent after rinsing and desiccation was distilled off under reduced pressure, and the powder of light brown was obtained. this -- column chromatography processing [-- support; -- silica gel eluate; -- toluene / ethyl-acetate (1/1)] -- after carrying out, it recrystallized [mixed solvent / of ethanol-water] and diacetyl object [general formula (** 1); R1=-COCH3 R2=-C6H4CH3 (p)] 7.11g of light brown prism-like ** was obtained.

Melting point 180.0-181.0 degrees C Elemental-analysis value (%)

C H N Fruit ** Value 66.09 4.72 4.64 C33H28F6N2 2O2 ***** -- calculated value 66.21 4.72 4.68

[0019] 7.11g of diacetyl objects acquired as mentioned above was dissolved in dioxane 170ml, 170ml of concentrated hydrochloric acid was added to this, and heating reflux was carried out for 4 hours. After cooling radiationally to a room temperature, iced water was filled with contents, and it extracted with toluene, and the solvent after rinsing and desiccation was distilled off under reduced pressure. The obtained crude material was *****ed from n-hexane and 2 of colorless needle shape crystal and 2-screw [4-N (p-tolylamino) phenyl] hexafluoropropane 4.76g was obtained.

Melting point 121.5-122.0 degrees C Elemental-analysis value (%)

C H N Fruit ** Value 67.70 4.74 5.42 C29H24F6N2 ***** -- calculated value 67.69 4.71 5.45 --

although the infrared-absorption-spectrum Fig. (KBr briquette method) of this compound was shown in drawing 3 -- 3400cm⁻¹ -- nuNH -- nuCF was accepted in 1-1 1320cm⁻¹ or 1175cm.

[0020] Example 4 [general formula (** 1); R1=R2-C6H4CH3 (p)]

1.03g [of secondary amine obtained in the example 3] and p-iodine toluene 1.75g, 1.11g of potassium carbonate, and 0.20g of copper powder were taken to nitrobenzene 20ml, and heating reflux was carried out under the nitrogen air current for 6 hours, carrying out azeotropy dehydration. After cooling





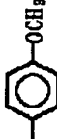

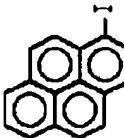
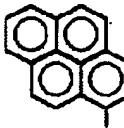

radiationally to the room temperature and carrying out filtration removal of the insoluble section, the nitrobenzene was distilled off under reduced pressure and residue was dissolved in toluene. The solvent after rinsing and desiccation was distilled off for the toluene layer under reduced pressure, and light brown oily matter was obtained. After carrying out column chromatography processing (support; silica gel eluate; toluene / n-hexane = 1/2) of this, it recrystallized [mixed solvent / of cyclohexane-ethanol] and 2 of non-color plate-like ** and 2-screw [4-N and N-bis(p-methylphenyl) aminophenyl] hexafluoropropane 0.91g was obtained.

Melting point 197.0-198.0 degrees C Elemental-analysis value (%)

C H N Fruit ** Value 74.34 5.21 3.90 C₄₃H₃₆F₆N₂ ***** -- calculated value 74.33 5.23 4.03 -- the infrared-absorption-spectrum Fig. (KBr briquette method) of this compound is shown in drawing 4 .

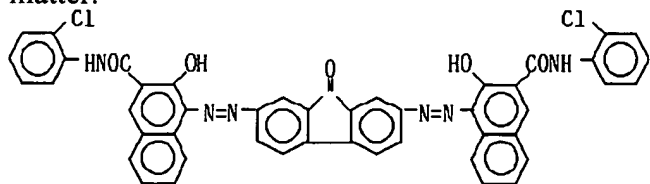
[0021] The aryl halide shown in Table 1 instead of p-iodine toluene used in five to example 7 example 4 was used, and also it reacted according to the example 4, and the tertiary-amine compound of this invention was obtained. A result is shown in Table 1.

[Table 1]

| 実施例No. | 使用したハロゲン化アリール | 一般式(1) | | 融点℃ (再結晶溶媒) | 元素分析値(%) 実測値(計算値) | | |
|--------|---|---|---|--|-------------------|----------------|----------------|
| | | R ₁ | R ₂ | | C | H | N |
| 5 |  |  |  | 173.0~174.5 (エタノール/ n-ヘキサン) | 74.06 (73.85) | 4.81 (4.85) | 4.30 (4.20) |
| 6 |  |  |  | 188.5~189.5 (トルエン/ n-ヘキサン) | 71.32 (71.05) | 4.94 (5.00) | 3.80 (3.86) |
| 7 |  |  |  | 217 ^{a)} (トルエン/ n-ヘキサン) | 80.27 (80.06) | 4.21 (4.42) | 2.96 (3.06) |

a) DTA吸熱ピーク温度

[0022] It is the following screw azo compound 7.5 section [** 3] as application charge generating matter.



And grinding mixing of the 0.5% tetrahydrofuran solution 500 of polyester resin [Byron 200 by Toyobo Co., Ltd.] section was carried out in the ball mill, the obtained dispersion liquid were applied with the doctor blade on vacuum-plating-of-aluminium polyester film, it seasoned naturally and the charge generating layer of about 1-micrometer thickness was formed. Next, the compound 1 section of an example 5 was dissolved in the resin solution of the polycarbonate resin [Teijin, Ltd. bread-making light K-1300] 1 section and the tetrahydrofuran 8 section as charge transportation matter, this solution was applied with the doctor blade on said charge generating layer, subsequently it dried for 5 minutes at 120 degrees C for 2 minutes by 80 degrees C, the charge transportation layer with a thickness of about 20 micrometers was formed, and the photo conductor was created.

[0023] Next, in order to investigate the sensibility in the visible region of the laminating mold electrophotography photo conductor obtained in this way, the electrostatic tracing paper testing device [Kawaguchi Electrical machinery Factory SP428 mold] was used for this photo conductor, and after having measured the surface potential V_m of a photo conductor (v) after performing -6kV corona discharge for 20 seconds and making it charged in a dark place, and leaving it in a dark place for 20 more seconds, surface potential V_o (v) was measured. Subsequently, tungsten lamp light was irradiated so that the illuminance on the front face of a photo conductor might be set to 4.5lux(es), and light exposure $E_{1/2}$ until V_o is set to one half (lux-sec) were measured.

$V_m = -1708V$ $V_o = -1553V$ $E_{1/2} = 3.33$ lux-sec

[Translation done.]